



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2710

Montana Soil

Highly Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. SRM 2710 is a highly contaminated soil that was oven-dried, sieved, and blended to achieve a high degree of homogeneity. A unit of SRM 2710 consists of 50 g of the dried material.

The certified elements for SRM 2710 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should not be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification of SRM 2710 is valid, within the measurement uncertainties specified, until **01 July 2015**, provided the SRM is handled in accordance with instructions given in this certificate (see Instructions for Use). This certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Stability: This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

Instructions for Use: A minimum sample weight of 250 mg (dry weight - see Instructions for Drying) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to achieve complete dissolution. If volatile elements (i.e., Hg, As, Se) are to be determined, precautions should be taken in the dissolution of SRM 2710 to avoid volatilization losses.

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills and J.S. Kane. Revision of this certificate was coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899

Certificate Issue Date: 28 October 1997*

30 Oct 92 (original certificate date); 23 Aug 93 (addendum added); 15 Aug 97 (strontium value revised)

*This revision reports editorial changes only.

Thomas E. Gills, Chief

Standard Reference Materials Program

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Jr., of the NIST Analytical Chemistry Division.

Instructions for Drying: When nonvolatile elements are to be determined, samples should be dried for 2 h at 110 °C. Volatile elements (i.e., Hg, As, Se) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture is to be made to the data for volatile elements before comparing to the certified values. This procedure ensures that these elements are not lost during drying. The weight loss on drying has been found to be in the range of 1.7% to 2.3%.

Source and Preparation of Material: The U.S. Geological Survey (USGS), under contract to the NIST, collected and processed the material for SRM 2710. The soil was collected from the top 10 cm (4 in) of pasture land located at Longitude 112° 47' and Latitude 46° 01' along Silver Bow Creek in the Butte, Montana area. The site is approximately nine miles east of the local Anaconda plant and 6.5 miles south of settling ponds that feed the creek. The creek periodically floods, depositing sediment with high concentrations of copper, manganese, and zinc at the collection site. The material was shoveled from a 6.1 m x 6.1 m (20 ft x 20 ft) area into polyethylene bags in cardboard cartons for shipment to the USGS laboratory for processing.

The material was spread on 30.5 cm x 61 cm (1 ft x 2 ft) polyethylene-lined drying trays in an air drying oven and dried for three days at room temperature. The material was then passed over a vibrating 2 mm screen to remove plant material, rocks, and large chunks of aggregated soil. Material remaining on the screen was deaggregated and rescreened. The combined material passing the screen was ground in a ball mill to pass a 74 µm screen and blended for 24 h. Twenty grab samples were taken and measured for the major oxides using x-ray fluorescence spectrometry and for several trace elements using inductively coupled plasma atomic emission analysis to provide preliminary assessment of the homogeneity of the material prior to bottling. The material was bottled into 50 g units and randomly selected bottles were taken for the final homogeneity testing.

Analysis: The homogeneity, using selected elements in the bottled material as indicators, was assessed using x-ray fluorescence spectrometry and neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the overall uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity is less than 2% for those elements for which homogeneity was assessed.

Certified Values and Uncertainties: The certified values are weighted means of results from two or more independent analytical methods, or the mean of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry used by two different laboratories employing different methods of sample preparation prior to measurement. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel [1] (NBS Journal of Research 87, 1982, pp. 377-385). The stated uncertainties include allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95% prediction interval and includes an allowance for systematic error among the methods used. In the absence of systematic error, a 95% prediction interval predicts where the true concentrations of 95% of the samples of this SRM lie. The certified values were corroborated by analyses from nine Polish laboratories cooperating on the certification under the direction of T. Plebanski and J. Lipinski, Polish Committee for Standardization, Measures, and Quality Control. The Polish laboratory work was supported by the Maria Skłodowska-Curie Joint Fund.

Table 1. Certified Values

Element	Mass Fraction (in %)			Element	Mass Fraction (in mg/kg)		
Aluminum	6.44	±	0.08	Antimony	38.4	±	3
Calcium	1.25	±	0.03	Arsenic	626	±	38
Iron	3.38	±	0.10	Barium	707	±	51
Magnesium	0.853	±	0.042	Cadmium	21.8	±	0.2
Manganese	1.01	±	0.04	Copper	2950	±	130
Phosphorus	0.106	±	0.015	Lead	5532	±	80
Potassium	2.11	±	0.11	Mercury	32.6	±	1.8
Silicon	28.97	±	0.18	Nickel	14.3	±	1.0
Sodium	1.14	±	0.06	Silver	35.3	±	1.5
Sulfur	0.240	±	0.006	Vanadium	76.6	±	2.3
Titanium	0.283	±	0.010	Zinc	6952	±	91

Noncertified Values: Noncertified values are provided for information only. An element concentration value is not certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (in %)	Element	Mass Fraction (in mg/kg)
Carbon	3	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7
		Strontium	330
		Thallium	1.3
		Thorium	13
		Tungsten	93
		Uranium	25
		Ytterbium	1.3
		Yttrium	23

Table 3. Analytical Methods Used for the Analysis of SRM 2710

Element	Certification Methods*	Element	Certification Methods*
Ag	ID ICPMS, RNAA, INAA	Mg	XRF1, ICP
Al	XRF1, XRF2, DCP, ICP	Mn	INAA, DCP, XRF2
As	RNAA, HYD AAS, ICP , INAA	Mo	ID ICPMS
Au	INAA , FAAS	Na	INAA, FAES
Ba	XRF2, FAES	Nd	ICP
Br	INAA	Ni	ID ICPMS, ETAAS, INAA
C	COUL	P	DCP, COLOR, XRF1, XRF2
Ca	XRF1, XRF2, DCP	Pb	ID TIMS, POLAR , ICP
Cd	ID ICPMS, RNAA	Rb	INAA
Ce	INAA , ICP	S	ID TIMS
Co	INAA , ETAAS, ICP	Sb	RNAA, ETAAS
Cr	INAA , DCP, ICP	Sc	INAA , ICP
Cs	INAA	Si	XRF1, XRF2, GRAV
Cu	RNAA, FAES, ICP	Sm	INAA
Dy	INAA	Sr	ID TIMS, INAA , ICP
Eu	INAA	Th	ID TIMS , INAA , ICP
Fe	XRF1, XRF2, DCP, INAA , ICP	Ti	XRF1, XRF2, DCP
Ga	INAA , ICP	Tl	ID TIMS , LEAFS
Hf	INAA	U	ID TIMS , INAA
Hg	CVAAS	V	INAA, ICP
Ho	INAA	W	INAA
In	INAA	Y	ICP
K	XRF1, XRF2, FAES, ICP	Yb	INAA
La	INAA , ICP	Zn	ID TIMS, ICP , INAA , POLAR

*Methods in **bold** were used to corroborate certification methods or to provide information values.

COLOR - Colorimetry; lithium metaborate fusion

COUL - Combustion coulometry

CVAAS - Cold vapor atomic absorption spectrometry

DCP - Direct current plasma atomic emission spectrometry; lithium metaborate fusion

ETAAS - Electrothermal atomic absorption spectrometry; mixed acid digestion

FAAS - Flame atomic absorption spectrometry; mixed acid digestion except for Au, leached with HBr-Br₂

FAES - Flame atomic emission spectrometry; mixed acid digestion

GRAV - Gravimetry; sodium carbonate fusion

HYD AAS - Hydride generation atomic absorption spectrometry

ICP - Inductively coupled plasma atomic emission spectrometry; mixed acid digestion

ID ICPMS - Isotope dilution inductively coupled plasma mass spectrometry; mixed acid digestion

ID TIMS - Isotope dilution thermal ionization mass spectrometry; mixed acid digestion

INAA - Instrumental neutron activation analysis

LEAFS - Laser enhanced atomic fluorescence spectrometry; mixed acid digestion

POLAR - Polarography

RNAA - Radiochemical neutron activation analysis; mixed acid digestion

XRF1 - Wavelength dispersive x-ray fluorescence spectrometry on fused borate discs

XRF2 - Wavelength dispersive x-ray fluorescence spectrometry on pressed powder

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REFERENCE

[1] Paule, R.C. and Mandel, J., *NBS Journal of Research* 87, 1982, pp. 377-385.

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.